Chapter 2 ~ Introduction

Chapter 2 provides detailed sampling and analysis procedures for "nutrient" parameters targeted in the Water Quality Surveys. These nutrients are:

- Nitrate+Nitrite Nitrogen
- Chloride
- Particulate Nitrogen
- Total Phosphorous
- Reactive Silica
- Total Dissolved Phosphorous
- Particulate Phosphorus
- Particulate Organic Carbon
- Dissolved Organic Carbon
- Calcium
- Magnesium
- Sodium

All samples for nutrient determination are collected with the Rosette sampling device as described in SOP LG 200, *Field Sampling Using the Rosette Sampler*. Because the Rosette is also used to collect samples for several non-nutrient parameters, SOP LG 200 also includes sample collection procedures for several physical, biological, and board chemistry parameters. Because the primary focus of Chapter 2 is on nutrient parameters, additional information regarding sample processing and analysis procedures for physical, biological, and board chemistry parameters collected with the Rosette using SOP LG 200 are provided in Chapters 3, 4, and 5.[1] Dissolved organic carbon generally is not included as a survey parameter but is determined for special studies, such as when hydrophobic organic compounds are a parameter of interest.

In the event that the Rosette is not operational, nutrient (and other associated parameters) samples are to be collected with SOP LG 201, *Niskin Bottle Hydrographic Line Sampling*.

Sample holding times for nutrient parameters are located in Appendix B, *Quality Assurance Project Plan for the Great Lakes Water Quality Surveys*, in Table 9-1, "Sample Preservation Methods, Holding Times, and SOPs."

Hard-copy data forms for documenting field and laboratory activities for the parameters included in this chapter are located in Appendix H.

***Note on Quality Control:** The following note describes the methods used by GLNPO and its contract/grantee laboratories to assure consistent quality of chemical analytical results. This manual is directed toward documenting and achieving a standard for the <u>current</u> program and does not necessarily reflect historical usage, since small changes are made as experience dictates.*

For almost all analyses, calibration standards are prepared or purchased according to the analytical SOPs. There is no control over these standards. It is presumed that care is taken in their preparation and their preparation is documented either on the label or in a log book with content, date of preparation, preparer and method of preparation.

The record of how well the analysis is generating precise and accurate results at any time is determined by analyzing quality control and quality assurance samples along with the regular

[1] Instructions for total suspended solids are provided in Chapter 3, phytoplankton and chlorophyll *a* are in Chapter 4, and turbidity, specific conductance, pH, total alkalinity, and dissolved oxygen are in Chapter 5.

samples from the environment. The control samples or standards are prepared independently and if possible by a different individual than the calibration standards.
The following definitions apply to the control standards:
Laboratory blanks - reagent water and reagent water dosed in the laboratory with the same amount of preservative as used to preserve the samples.

Control standard high level and control standard low level - laboratory stoichiometric preparations of reagent material near the upper limit and the lower limit respectively of the concentrations found in the environmental samples. These control standards are dosed with whatever preservatives are used for preserving the environmental samples.

The following definitions apply to the quality assurance samples:

Field blanks: reagent water treated the same as the samples removed from the Niskin bottles.

Laboratory spikes: environmental samples to which a measured amount of concentrated analyte is added. The purpose of spiking samples is to determine if a substance is present in the sample that may interfere with the analytical procedure, either accentuating or diminishing the response of the analyte to the analytical conditions. The same analytical procedures have been used by GLNPO for over 20 years and spikes were a part of the routine analytical procedure for the first 10 years. Throughout that first ten years, there was no indication of any type of interference in the lake water to these analytical procedures. At present GLNPO does not routinely analyze spiked samples.

Field duplicates: a second sample using a different Niskin bottle, but collected at virtually the same time and place as the original sample.

Laboratory duplicate: a second aliquot from the same cubitainer or Niskin bottle processed like the original sample. It is separately filtered and/or preserved in the field.

Instrument replicate: replicate instrument readings are not documented by GLNPO since experience has shown that detectable differences are insignificant relative to the other duplicates.

Field blanks, laboratory duplicates, and field duplicates are labeled and tracked with pre-designated sampleIDs. High and Low Control Standards and those laboratory blanks that require addition of a preservative to bring them to the same relative condition as the environmental samples are documented when they are prepared and given a unique sampleID. That same sampleID is assigned each time that they are analyzed. This unique sampleID is associated with and used only for that particular container of that preparation. If that container is refilled with material from a bulk container, a different sampleID is assigned to the new material even though they both originated from the same bulk container. This is a deviation from past procedure in which a unique sampleID was assigned to the High and Low Control standards and the laboratory blanks for each batch of samples but the same sampleID was used for different standards, i.e., chloride, nitrate and phosphorus, so that different material had the same sampleID, and the same material had different sampleIDs from batch to batch.

In an effort to determine the shelf-life of samples using our present system and/or to add some assurance of comparability to our data, we are beginning a system of archiving routine samples for re-analysis after 1, 2, 3, 4, and 5 years. This will be done only with the spring samples for nutrient analysis. We will retain 5 analyzed samples from each lake each cruise from each nutrient group. One will be re-analyzed the following year, one the second year, etc. After five

years, five archived samples will be analyzed with each lake cruise, each with a different age (i.e., 1, 2, 3, 4, and 5 years old). We will have in storage 15 samples (5+4+3+2+1) for each lake for each analysis. Each archived sample will be analyzed only twice; once the year of collection and again after a one to five year delay.

Revision Sheet

Method Number	SOP Title	New Revision Number	Description of Changes	Updated Revision Number and Date on SOP/TOC			Date	Initials of Person Responsible for Changes	Approval Signature
				Cover	Footers	TOC			